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Indian Standard

RESINS FOR PAINTS — METHODS OF SAMPLING AND TEST

PART 7 SPECIAL TEST METHODS FOR ACRYLIC AND
VINYL ACETATE RESINS AND EMULSIONS

(Second Revision)

भारतीय मानक

रंग रोगनों के लिए रेज़िनों के नमूने लेने की और परीक्षण-पद्धतियाँ

भाग 7 एकलिक और बिनाइल ऐसिटेट रेज़िनों और इमल्सनों की विशेष परीक्षण-पद्धतियाँ

(दूसरा पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (Part 7) (Second Revision) was adopted by the Bureau of Indian Standards on 16 August 1989, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1952 covering methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for methods of sampling and test for natural and synthetic resins was published as Part 2 of the above standard in 1971. These two parts were amalgamated and revised in 1976. This revision has been necessitated as more and more newer synthetic resins like polyamides, polyvinyls and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee felt it appropriate to publish this standard in various parts, as indicated below:

- Part 1 General test methods
- Part 2 Special test methods for alkyd resins
- Part 3 Special test methods for phenolic resins
- Part 4 Special test methods for epoxy resins
- Part 5 Special test methods for polyamide resins
- Part 6 Special test methods for amino resins
- Part 7 Special test methods for acrylic and vinyl acetate resins and emulsions

In preparing this standard assistance has been derived from ISO 2115-1976 'Plastics — Aqueous dispersions of polymers and copolymers — Determination of white point temperature and minimum film-forming temperature', issued by the International Organization for Standardization.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

RESINS FOR PAINTS — METHODS OF SAMPLING AND TEST

PART 7 SPECIAL TEST METHODS FOR ACRYLIC AND VINYL ACETATE RESINS AND EMULSIONS

(Second Revision)

1 SCOPE

1.1 This standard prescribes the special test methods for acrylic and vinyl acetate resins and emulsions.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
323 : 1959	Specification for rectified spirit (revised)
1303 : 1983	Glossary of terms relating to colorimetric and volumetric analysis (second revision)
2316 : 1989	Methods of preparation of standard solutions for paints (second revision)
6667 : 1972	Glossary of terms used in synthetic resin industry

3 TERMINOLOGY

3.1 The definitions given in IS 1303 : 1983 and IS 6667 : 1972 in addition to the following shall apply.

3.1.1 Minimum Film-Forming Temperature

The limiting temperature above which a continuous homogeneous film without cracks is formed.

3.1.2 White-Point Temperature

The limiting temperature below which an opaque mass, and above which a transparent film, is formed.

4 SAMPLING

4.1 Representative samples of the product shall be drawn as prescribed in 3 of Part 1 of this standard.

5 IDENTIFICATION

5.1 Acrylic resins are clear transparent and mostly water white solutions, while emulsions are milky white viscous liquids.

6 DETERMINATION OF FREE MONOMER CONTENT

6.1 Determination of Free Acrylate Monomer Content in Acrylic Resins

6.1.0 Outline of the Method

Determination of these residual monomers is based on reaction with morpholine or piperidine, which adds to the double bond, forming a tertiary amine. Subsequently, treatment with acetic anhydride converts the excess secondary amine to an amide and the tertiary amine can be titrated using standard alcoholic hydrochloric acid.

6.1.1 Reagents

6.1.1.1 Hydrochloric acid, 0.5 N in ethyl alcohol.

6.1.1.2 Methyl orange-xylene cyanol indicator, 0.1 percent. Dissolve 0.25 g of methyl orange and 0.15 g of xylene cyanol FF in 50 ml of rectified spirit (see IS 323 : 1959) and dilute to 250 ml with water.

6.1.1.3 Acetic acid, 50 percent aqueous solution.

6.1.1.4 Acetonitrile (methyl cyanamide)

6.1.1.5 Morpholine

6.1.2 Procedure

Dissolve 5 g of the sample in 25 ml of acetonitrile, add 10 ml of morpholine and 7 ml of 50 percent aqueous acid. After standing for 1 hour, 25 ml of acetonitrile and 20 ml of acetic anhydride are added. The mixture is cooled to room temperature and titrated with standard alcoholic hydrochloric acid solution using methyl orange-xylene cyanol indicator. A blank containing a weighed sample of resin with all other reagents except morpholine is run under parallel condition.

6.1.3 Calculation

Acrylic monomer expressed as methyl acrylate, percent by mass
$$= \frac{(V_1 - V) \times N \times M_w}{10 \times M}$$

where

V_1 = volume in ml of standard hydrochloric acid used for blank,

V = volume in ml of standard hydrochloric acid used for sample,

N = normality of standard hydrochloric acid,

M_w = molecular weight of methyl acrylate, and

M = mass in g of sample taken for test.

6.2 Determination of Free Vinyl Monomer Content in Vinyl Acetate Resins

6.2.0 Outline of the Method

Using excess bromine and back titrating the bromine with standard sodium thiosulphate solution, vinyl free monomer in the resin is determined.

6.2.1 Reagent

6.2.1.1 Bromine solution

Dissolve 60 g of potassium bromide and 3.8 g of bromine in water and make up to 1000 ml with water.

6.2.1.2 Potassium iodide solution, 15 percent.

6.2.1.3 Standard sodium thiosulphate solution, 0.1 N (see IS 2316 : 1989).

6.2.2 Procedure

Weigh accurately around 10 g of the resin into a 250-ml flask containing 50 ml of water. Shake the mixture. Add measured quantity of excess bromine solution and 20 ml of 15 percent potassium iodide solution. Titrate the excess bromine solution against standard sodium thiosulphate solution to faint yellow colour. For a blank determination, repeat the procedure using water instead of resin.

NOTE — Acrylates and maleates do not interfere in the estimation of free vinyl monomer.

6.2.3 Calculation

Free vinyl monomer
expressed as vinyl
acetate, percent
by mass

$$= \frac{(V_1 - V) \times N \times M_w}{20 \times M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used for blank,

V = volume in ml of standard sodium thiosulphate solution used for sample,

N = normality of standard sodium thiosulphate solution,

M_w = molecular weight of monomer vinyl, and

M = mass in g of the sample taken for test.

6.3 Determination of Free Monomer Content in Emulsions

6.3.0 Outline of the Method

This method involves the addition of bromine to the unsaturated free monomer and excess of bromine is estimated iodometrically.

6.3.1 Reagents

6.3.1.1 Methanol containing 100 ppm of *p*-tert-butyl catechol

6.3.1.2 Standard potassium bromide-potassium bromate solution 0.1 N

Dissolve 2.784 g of potassium bromate and 10.0 g of potassium bromide in water and dilute to 1000 ml.

Standardize with 0.1 N sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) solution in presence of an excess of potassium iodide and sulphuric acid.

6.3.1.3 Sulphuric acid, 18 percent.

6.3.1.4 Potassium iodide solution, 10 percent.

6.3.1.5 Standard sodium thiosulphate solution, 0.1 N (see IS : 2316 : 1989).

6.3.1.6 Starch indicator, 1 percent in water.

6.3.2 Procedure

Pipette out 25 ml of the resin into 250-ml joint flask, rinse the pipette with 25 ml of alcohol water and add the rinsings to the flask. Add 25 ml of methanol containing 100 ppm *p*-tert-butyl catechol. Connect the ground joint flask to the distillation apparatus with ground joints and distil the mixture. Collect the first 25 ml of distillate in a 250-ml iodine flask, rinse the condenser with 20 ml of methanol containing 100 ppm of *p*-tert-butyl alcohol and add the rinsings to the jointed flask. From the burette add 20 ml of 0.1 N potassium bromide potassium bromate solution. Cool the solution to 30°C. Rapidly add 15 ml of 18 percent sulphuric acid, stopper the flask, shake it, and add distilled water as a vapour seal. Allow the bottle to stand for 60 seconds. If no yellow colour remains, add successive two 10-ml portions of potassium bromide potassium bromate solution until a slight yellow colour persists for 60 seconds after the addition. Make the additions by drawing the standard solution from the burette into the space between the stopper and the neck of the flask by lifting the stopper so that the solution enters the flask around the stopper. Wash the space between the stopper and the neck of the flask with distilled water in the same manner and seal with water. After 60 seconds of the final bromide bromate addition, add 10 ml of 10 percent potassium iodide solution to the space between the stopper and the neck of the flask and lift the stopper to allow the solution to enter the flask around the stopper. Shake the flask and contents. Titrate the liberated iodine with 0.1 N standard sodium thiosulphate solution to a faint yellow continue the titration with sodium thiosulphate solution until the solution is clear. For a blank determination repeat the procedure using water instead of resin.

6.3.3 Calculation

$$\text{Free monomer, percent by mass} = \frac{(V_1 - V) N X}{M}$$

where

V_1 = volume in ml of sodium thiosulphate solution used for blank,

V = volume in ml of sodium thiosulphate solution used for sample,

N = normality of standard sodium thiosulphate solution,

M = mass in g of resin taken for test, and

X = molecular weight of the monomer/20.

7 DETERMINATION OF STABILITY OF EMULSIONS

7.0 Two methods are prescribed for determination of stability of emulsions, namely, mechanical method and electrolyte method.

7.1 Mechanical Method

7.1.0 Outline of the Method

A sample of emulsion is subjected to mechanical shear by the use of a high-speed stirrer and the extent of instability noted. The amount of coagulum formed after a given time of agitation is considered a measure of instability.

7.1.1 Apparatus

7.1.1.1 Tester

The test apparatus shall consist of a vertical shaft high-speed stirrer capable of maintaining a speed of 1470 ± 21 rad/s for the duration of the test. The stirrer shaft shall be approximately 6.3 mm in diameter at its lower end at the point of attachment of the agitator disk and may taper upward for greater strength. It shall be of sufficient length to reach conveniently to the bottom of the test bottle. The shaft shall run with not more than 0.25 mm out of true at the speed specified.

7.1.1.2 Agitator disk

The agitator itself shall consist of a polished stainless steel disk 36.12 ± 0.03 mm in diameter and 1.58 ± 0.05 mm in thickness having a threaded stud at its exact centre for attachment to the centre of the lower end of the stirrer shaft.

7.1.1.3 Test bottle

The test bottle shall be a flat bottom cylindrical glass container 57.8 ± 1 mm in inside diameter by approximately 12.7 cm in height, and having a wall thickness of approximately 2.3 mm.

7.1.1.4 Bottle holder

The bottle holder shall be so constructed that the bottle may be conveniently lowered and raised to the exact specified position with relation to the shaft and agitator. The position of the test bottle shall be such that the axis of the stirrer shaft is concentric with the axis of the bottle and that the bottom of the agitator disk is 12.7 ± 2 mm from the bottom inside of the bottle.

7.1.1.5 Stainless steel screen

180 micron sieve with 0.180 ± 0.009 mm opening and 0.131 ± 0.01 mm wire diameter. The screen shall be cut to fit into the seat of a pipe union having inside diameter of about 5 cm.

7.1.1.6 Potassium oleate, 2 percent aqueous solution.

7.1.2 Procedure

Reduce the viscosity of emulsion below 350 mpa.s (cps) unless the solids are below 10 percent relative.

7.1.2.1 Strain the emulsion through a 180 micron sieve and weigh 50 ± 0.5 g of the strained emulsion into the test bottle. Place the bottle containing the emulsion in the holder of the stability tester.

7.1.2.2 Start the tester and agitate for exactly 30 minutes (or other agreed-upon time) at 1470 ± 21 rad/s (14000 ± 200 rev/min). The time of agitation should be adjusted so that the temperature of emulsion does not increase to more than 60°C. Remove the sample immediately to avoid any filming and rinse the agitator shaft and disk with distilled water. Strain the emulsion through a tared 180-micron screen that has previously been wetted with a 2 percent aqueous solution of a surfactant such as potassium oleate. Finally, flush the screen with the same solution and then follow with a rinse of water. Dry the screen and coagulum in an oven at $100 \pm 2^\circ\text{C}$ to constant mass (approx 15 minutes). Cool and weigh again to ± 0.001 g.

7.1.3 Calculation

Calculate the percent coagulum content as follows:

$$\text{Coagulum content, percent} = \frac{(A - B)}{m} \times 100$$

where

A = mass of tared screen plus coagulum,

B = mass of tared screen, and

m = mass of the strain emulsion.

7.2 Electrolyte Method

7.2.0 The stability of emulsions in the presence of a known quantity of aluminium sulphate solution is known as electrolyte stability.

7.2.1 Reagents

7.2.1.1 Aluminium sulphate solution. Dissolve 50 g crystalline aluminium sulphate in 50 ml of water to give a 50 percent electrolyte solution.

7.2.2 Procedure

Take 100 g of emulsion in the 250 ml beaker and introduce the glass rod to be used as agitator.

7.2.2.1 Take the aluminium sulphate solution in the burette and start adding in drops to the emulsion under stirring. When the emulsion coagulates stop addition.

7.2.2.2 The number of ml of electrolyte solution required to coagulate 100 g of emulsion is known as electrolyte stability.

7.2.3 Calculation

Electrolyte stability = number of ml of electrolyte solution (50 percent) required to coagulate.

8 DETERMINATION OF WHITE POINT TEMPERATURE AND MINIMUM FILM-FORMING TEMPERATURE

8.0 Principle

Application of a suitable temperature gradient between a hot source and a cold source on a metal plate (chromed aluminium) which may be either perfectly levelled and smoothened or which may be channelled from the cold source to the hot source. Spreading out of one or more films of the dispersion on the level plate, or filling of the channels (starting at the end nearer the hot source) section of the dispersion. Drying with a current of moisture-free air and determination of the temperature at which the coalesced (transparent) section of the dispersion meets the uncoalesced (white) section.

8.1 Apparatus

8.1.1 Test Apparatus (see Fig. 1)

Consisting essentially of a rectangular plate, of chromed aluminium, whose surface may either be perfectly level and polished, or may contain channels, four number of 0.3 mm deep.

8.1.1.1 At one end of the plate is a rheostat-controlled electrical resistance, which constitutes the hot source. At the opposite end is the cold source, consisting either of an insulated container in which refrigerant is placed or of a coil inserted into the end of the plate and through which a refrigerant mixture is circulated.

8.1.1.2 Evenly spaced along the plate are holes into which thermometers can be inserted to measure the temperature gradient of the plate when equilibrium is reached. The first hole is positioned at the cold end of the plate at right angles to the beginning of the channels, if the plate has them.

8.1.1.3 Provision should be made for placing a glass cover above the plate, leaving a space through which a slight current of dry air (dried, for example, by passing through a column packed with calcium chloride) can be directed at room temperature, from the cold end to the hot end.

8.1.2 Devices for Temperature Measurement

Having an accuracy of in the range of $\pm 10^\circ\text{C}$; for instance, a mercury-in-glass thermometers, thermocouples, surface thermometer, etc.

8.1.3 Film Spreader

Made of stainless steel, capable of producing simultaneously and successively either films in the channels (see Fig. 2), or film about 0.1 mm thick and 20 to 25 mm wide on plates without channels.

8.2 Procedure

8.2.1 Establishment of Temperature Gradient

Put in place the devices for temperature measurement. Use the hot source and cold source to establish a suitable temperature gradient for the dispersion being tested so that:

- the 'white point' temperature to be determined occurs in the centre section of the plate; and
- the temperature range of the two extreme thermometers is between 20°C and 40°C and remains constant during the test.

NOTE — As far as possible, the temperature gradient should be linear, that is, the temperature differences between successive devices for temperature measurement should all be of the same order.

8.2.1.1 Wait until thermal equilibrium is reached, that is, until the devices for temperature measurement show very little further variation.

8.2.2 Determination

When using a level plate, spread out the dispersion with the film spreader, starting from the end with the highest temperature. In the case of a channelled plate, pour into the channels, at the end of the plate with the highest temperature, a quantity of dispersion slightly in excess of the total capacity of the channels. Spread this out using the film spreader. In both cases, replace the glass cover so as to obtain a sealed chamber and circulate through this, from the cold end to the hot end, a current of dry air at room temperature and at a low, constant rate of flow.

8.2.2.1 Wait until there is complete separation of the section, transformed into film (transparent) and the untransformed section (opaque). Measure the distance between the first point of temperature measurement and the line of separation. Note the temperatures indicated by the various devices for temperature measurement inserted into the plate and plot a graph using distance intervals between devices as the abscissae and the plate temperatures as the ordinates.

NOTE — If the temperature gradient is linear, the curve obtained is a straight line and in practice it is then unnecessary to plot a graph.

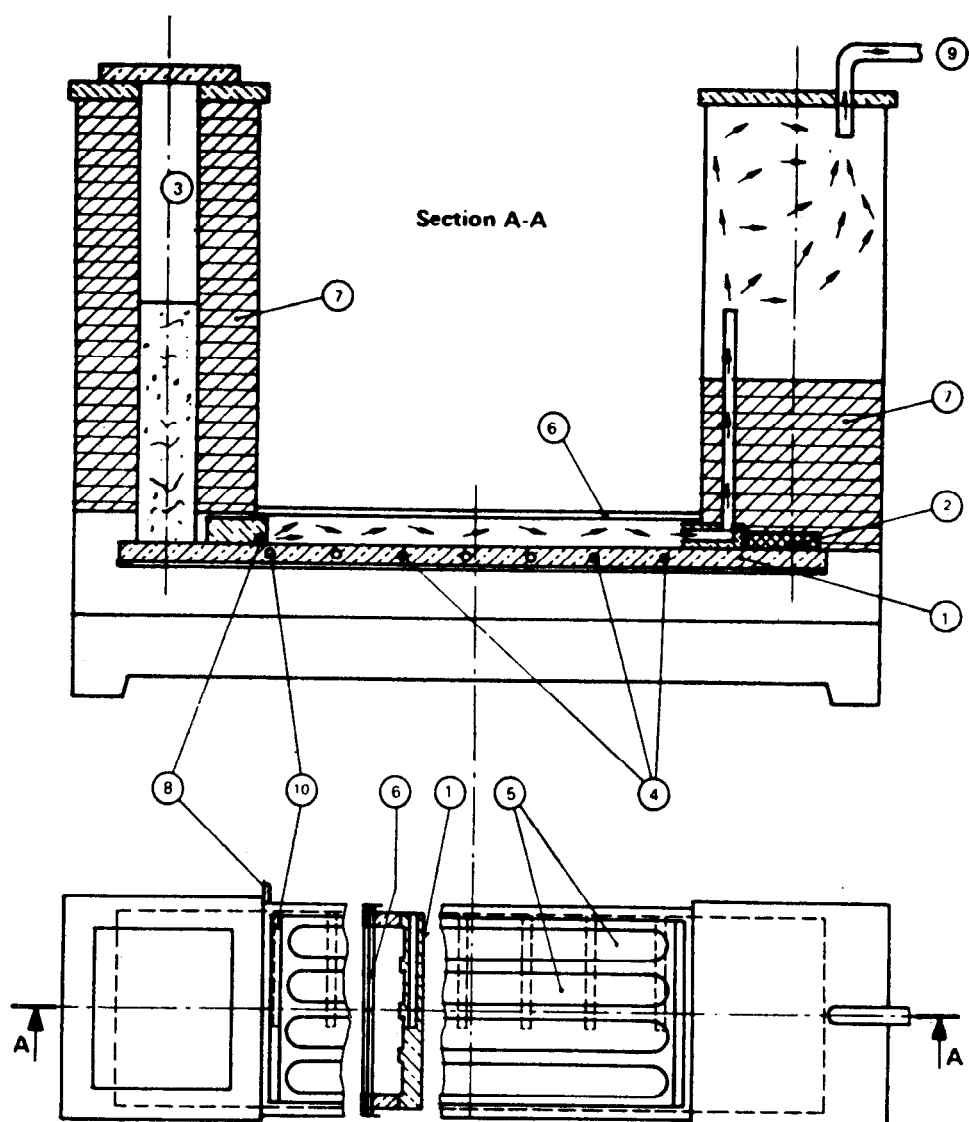
8.2.2.2 Using the graph, determine the 'white point' temperature of the dispersion being tested. If a surface thermometer is used, the 'white point' temperature can be determined directly.

8.2.2.3 Determine the lowest temperature where a continuous, homogeneous film without cracks is formed. This is the minimum film-forming temperature and record.

8.3 Test Report

The test report shall give the following information:

- The identification characteristics of the dispersion tested;
- The type of testing apparatus;
- The value of the 'white point' temperature in degree Celsius; and
- The minimum film-forming temperature in degree Celsius.

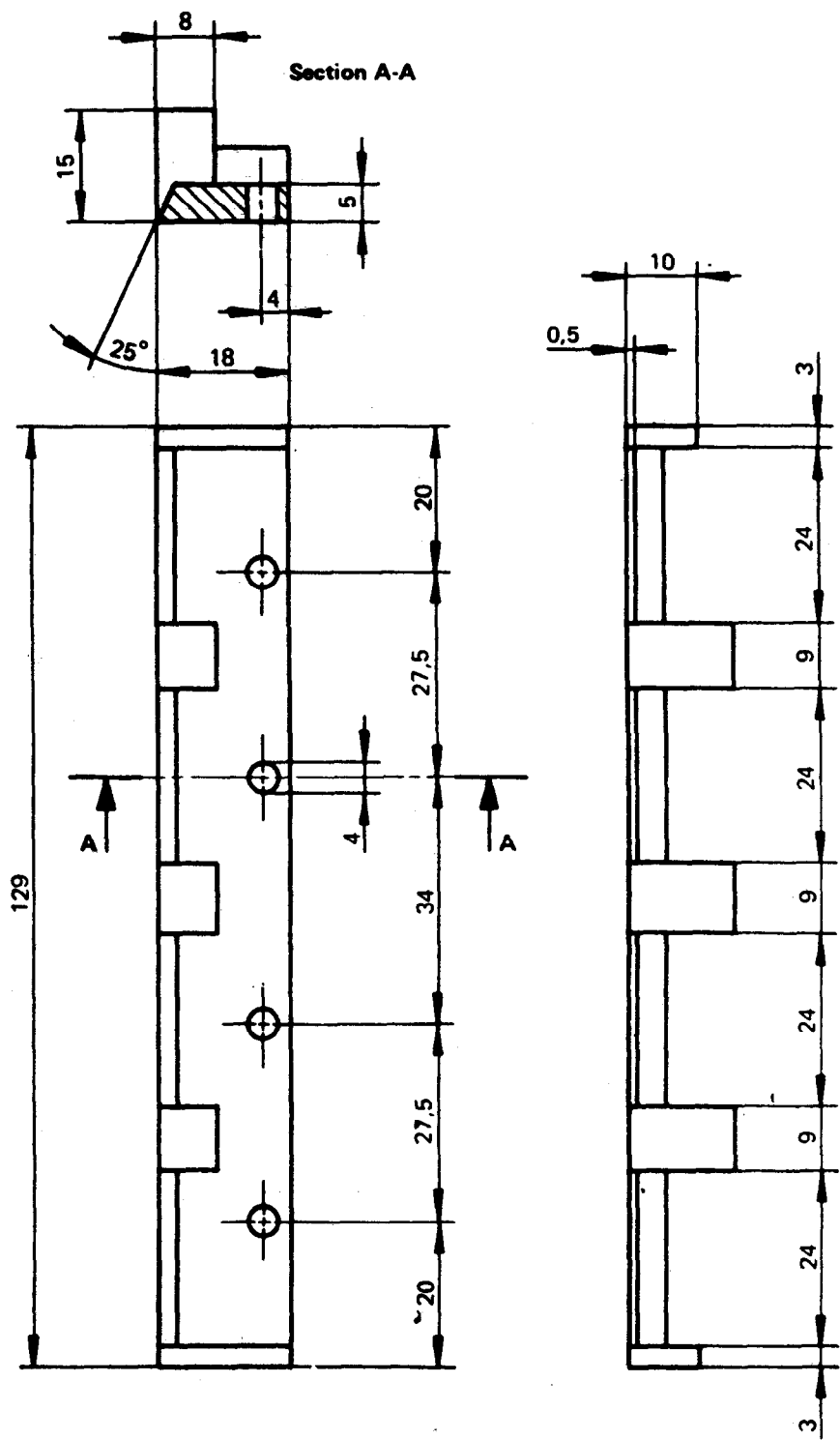


KEY

- | | |
|---|---------------------------|
| 1 Rectangular metal plate | 6 Glass cover |
| 2 Electrical resistance | 7 Insulating material |
| 3 Reservoir for refrigerant mixture | 8 Dry air inlet |
| 4 Holes for thermometers (diameter : 5 mm) | 9 Air outlet |
| 5 Channels in the upper surface of the plate (depth : 0.3 mm) | 10 First thermometer hole |

NOTE — The plate in this diagram is channelled but this feature is not essential.

FIG. 1 DIAGRAM OF APPARATUS



All dimensions in millimetres.

FIG. 2 EXAMPLE OF FILM SPREADER FOR A CHANNELLED PLATE

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